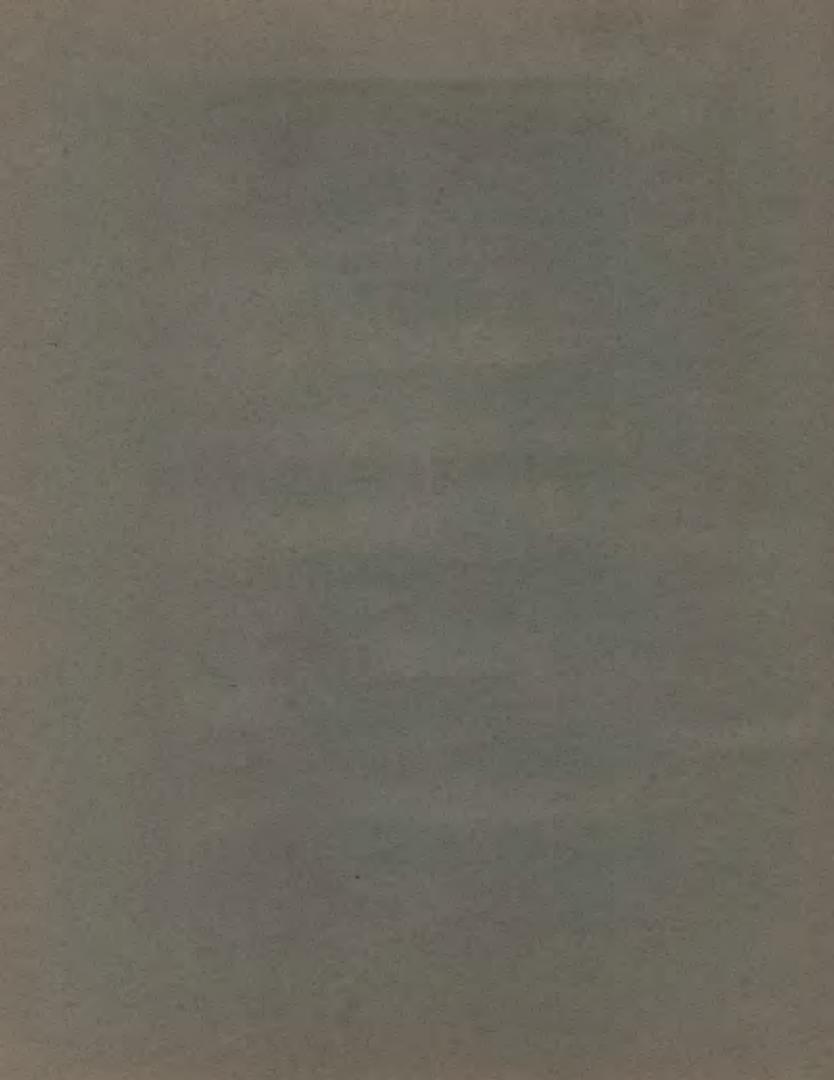
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# SOME STANDARD THERMAL DEHYDRATION CURVES OF MINERALS

GEOLOGICAL SURVEY PROFESSIONAL PAPER 197-E



# UNITED STATES DEPARTMENT OF THE INTERIOR Harold L. Ickes, Secretary

GEOLOGICAL SURVEY W. C. Mendenhall, Director

Professional Paper 197-E

# SOME STANDARD THERMAL DEHYDRATION CURVES OF MINERALS

BY

P. G. NUTTING

Shorter contributions to general geology, 1941-42

(Pages 197-217)



UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON: 1943

### CONTENTS

Abstract	
Introduction	
Thermal dehydration determinations	
Materials showing slight curvature variation.	
Hydrous oxides and zeolites	
Hydrous oxides and related silicates	
Kaolin group	
Montmorillonites	
Montmorillonite group	
Chlorites and related silicates	
Micas	
Carbonates:	
ILLUSTRATIONS	
ILLUSTRATIONS	-
ILLUSTRATIONS  ———— FIGURE 14. Curves of slight curvature variation	
ILLUSTRATIONS  ————  FIGURE 14. Curves of slight curvature variation  15. Curves for hydrous oxides and zeolites	
ILLUSTRATIONS  ————  FIGURE 14. Curves of slight curvature variation	·
FIGURE 14. Curves of slight curvature variation  15. Curves for hydrous oxides and zeolites  16. Curves for hydrous oxides and related silicates	
FIGURE 14. Curves of slight curvature variation  15. Curves for hydrous oxides and zeolites  16. Curves for hydrous oxides and related silicates  17. Curves for the kaolin group	
ILLUSTRATIONS  ILLUSTRATIONS  FIGURE 14. Curves of slight curvature variation  15. Curves for hydrous oxides and zeolites  16. Curves for hydrous oxides and related silicates  17. Curves for the kaolin group  18. Curves for montmorillonites  19. Curves for the montmorillonite group  20. Curves for chlorites and related silicates	
ILLUSTRATIONS  ILLUSTRATIONS  FIGURE 14. Curves of slight curvature variation  15. Curves for hydrous oxides and zeolites  16. Curves for hydrous oxides and related silicates  17. Curves for the kaolin group  18. Curves for montmorillonites  19. Curves for the montmorillonite group	

#### SOME STANDARD THERMAL DEHYDRATION CURVES OF MINERALS

By P. G. NUTTING

#### ABSTRACT

The curve representing the change in weight of a mineral as its temperature is raised is a well-recognized characteristic. Many individual curves have been obtained, but no sets of standard curves for reference have been published. From about 250 such curves obtained by the writer in the past 10 years, 73 of the more significant, arranged in 9 groups, are here presented.

Weights were obtained on a 1- or 2-gram sample, crushed to 150 mesh (0.1 millimeter), in a platinum crucible suspended in a vertical electric furnace from one arm of a balance mounted over the furnace. After reaching constant weight at one temperature, the temperature was advanced about 50° C. The curves refer to average room humidity, about 0.6 saturation—that is, to a vapor pressure of about 15 millimeters.

Some classification according to material has been attempted, but this is incidental to the grouping of curves according to their form. The groups of curves are as follows: (1) Curves lacking in decided shoulders and referring to such substances as silica gel, powdered quartz, volcanic ash, diatoms, and montmorillonite residue (fig. 14). (2) Hydrous oxides, such as opal, allophane, hydrated alumina, and sepiolite, and three zeolites (fig. 15). (3) Hydrous oxides and related silicates, including gibbsite, diaspore, limonite, and geothite (fig. 16). (4) The kaolin group of minerals—anauxite, kaolinite, and dickite (fig. 17). (5) Normal pure unweathered montmorillonite, the same material leached and oxidized by weathering, bentonites high in alkali, and fuller's earth (fig. 18). (6) The montmorillonite group, with halloysite, illite, and saponite (fig. 19). (7) Chlorites and related silicates, including serpentine, picrolite, clinochlore, penninite, paligorskite, vermiculite, and glauconite (fig. 20). (8) The micas and other minerals characterized by curves with rounded high-temperature shoulders, such as pyrophyllite, talc, tremolite, and beryl (fig. 21). (9) Five carbonates and hydrous carbonates (fig. 22).

Significant features of all these curves are discussed in the text, and the numerical data read from the curves are tabulated.

#### INTRODUCTION

The determination of water in minerals at two different temperatures is routine in chemical analysis. For many substances the loss in weight on heating to 110° C. is the free water, and the loss between 110° C. and ignition temperatures (800°–1000° C.) is the essential or crystalline water. Some minerals contain water in at least three different forms, and these forms can be distinguished and evaluated only when weights are determined at a whole series of temperatures so that a complete curve representing loss of water may be drawn through them. The clay minerals, zeolites, and hydrous oxides are conspicuous examples. With a series of such curves at hand for reference, the proper temperatures for water determination may often be

chosen. The weight-temperature curve of an unknown mineral sometimes serves to identify it when chemical and petrographic analyses cannot. The curve for a mixture of minerals may help to identify its constituents. With 250 such curves accumulated by the writer during the past dozen years, it seems desirable to select and publish the more significant ones to serve as the nucleus of a more comprehensive collection later to be available for reference. Many similar observations have been published, but no classified collection has been attempted.

In the lower range of temperature, weight curves vary with the humidity, an effect which vanishes above about 160° C. Determining weight-temperature curves (at constant humidity) and weight-humidity curves (at constant temperature) below 160° C. requires special and much more complicated apparatus than that used in this work. The curves here presented are for an average room humidity of about 60 percent, obtained by heating the specimen in a small open plat;num crucible in an electric furnace. Temperatures are read with a platinum, platinum-rhodium thermccouple projecting into the furnace. There may be some uncertainty in the details of curves that have abrupt breaks downward. To obtain such details with a greater degree of accuracy would require temperature control and measurement to within 2°, but ordinarily the furnace temperature is sufficiently constant.

To obtain a complete dehydration curve requires weighings at 10 to 20 points not more than 50° apart. Instead of removing the crucible and specimen from the furnace, cooling in a desiccator, and weighing, it seemed advisable to suspend the crucible permanently in the furnace and to make weighings about twice a day on a balance mounted over the furnace. The specimen is not disturbed during the whole run. The correction for air density is roughly 4 milligrams per gram for 800° C. A period of about 2 weeks is required to complete the average curve, but the actual working time required is only 5 minutes twice a day to keep two furnaces in operation. Very uniform temperatures throughout the furnace are obtained by using a wellfitted covering, with only a small hole for the suspension wire. Fairly constant furnace temperatures are obtained on the regular power line by using lamp rhecstats as ballast, the lamps being replaced by tubes containing iron wire in hydrogen, such as are now in commercial use for radio work. The large temperature coefficient of resistance of these tubes so reduces the effects of line voltage fluctuations that the furnace temperatures seldom vary as much as 2° C.

The sample is ground just fine enough to pass a 150-mesh (0.1-mm.) sieve. Coarser grains are slow in reaching constant weight, and much finer material fails to give a true curve on account of the increased proportion of broken bonds. Samples fresh from the ground are selected and dried just sufficiently to permit grinding. Prolonged room drying, storage in a saturated atmosphere, dispersion in water, etc., profoundly affect some clay and soil curves below 300° C.; dispersion in water or very fine grinding removes the high-temperature shoulder from some clay curves. The effects of electrodialysis and of treatment with water, acids, and alkalies is a field of research in itself.

Most clays and soils and many minerals, or at least their dehydration products, change little in weight in the range 700°-900° C. This constant weight is therefore taken as a base weight rather than the weight at 110° C. or at room temperature, where the weights of most clays vary widely with both humidity and temperature. Above 900° C. sintering is common and reactions may occur. In these curves interest centers on the shoulder usually identified with the loss of hydroxyl and of lattice structure and on the sloping portion leading up to it, where the slope is usually related to the loss of water that was firmly adsorbed as ions.

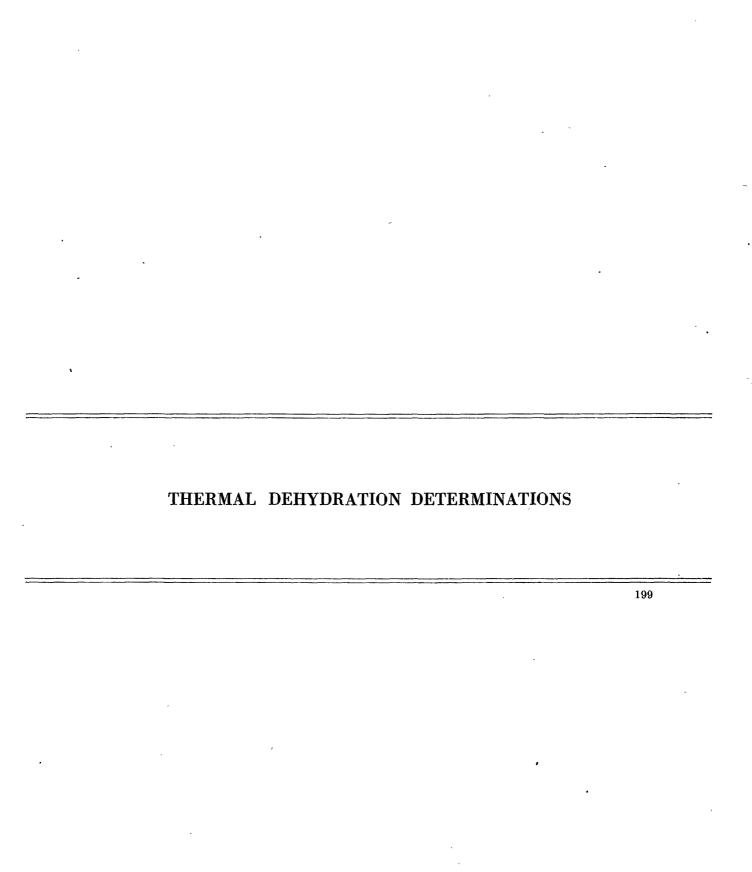
Oxidation of iron from the ferrous to ferric condition (usually at 200°-300° C.) involves a small negative

correction. Organic matter is oxidized at a low temperature. The decomposition of carbonates must of course be allowed for, if they are present. So far as known, most of the specimens studied were free from carbonates, but weight-temperature curves for a few known carbonate minerals are included in the last group (fig. 22) for comparison and reference. No extensive study of the thermal decomposition of carbonates has been attempted, as this field has been largely covered by other investigators.

#### THERMAL DEHYDRATION DETERMINATIONS

A number of representative weight-temperature determinations are reproduced in the accompanying diagrams (figs. 14-22). These are discussed in the text, and the numerical data read from the curves are given in tables. Some classification according to material has been attempted, but this is incidental to the grouping of curves according to their form.

Shoulders on a thermal decomposition curve are caused by rapid variation (with temperature) of the energy of association of some volatile constituents with the residual material. Conversely, the absence of such shoulders means either no variation in energy or variation changing only slowly. No mineral has been found whose thermal dehydration curve shows constant energy of association from  $20^{\circ}$  to  $800^{\circ}$  C., but many such curves are found to consist of only three straight lines when log (water) is plotted against the reciprocal of the absolute temperature. The slope of the curve thus plotted gives the energy of association in calories per mole divided by the constant R=1.9864.



#### MATERIALS SHOWING SLIGHT CURVATURE VARIATION

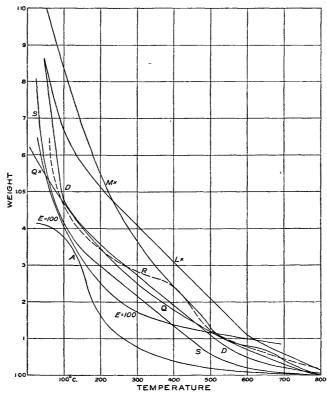


FIGURE 14.—Curves of slight curvature variation.

- Q, Powdered quartz (×10).
- E. Curve of constant energy.
- S, Amorphous silica.
- A. Volcanie ash.
- M, Nearly amorphous material having the composition of montmorillonite (half scale).
- R, Isotropic silica residue remaining after extraction of bases from a pure bentonite.
- D, Diatoms
- L, Synthetic magnesium silicate "Magnesol" (half scale).

Curve Q: The smoothest curves found are those given by finely powdered anhydrous crystalline minerals (quartz, feldspars). Here there is an average adsorption of H and OH on broken surfaces or chemical bonds of a wide variety. Curve Q, exaggerated 10 times, is for quartz ground to pass a 300-mesh sieve (0.05 mm. opening) such that the specific surface is about 560 sq. cm. per gram. Water decreases from 0.5 percent at 80° C. to 0.07 percent at 600° C. Energy of association increases steadily, being 38 calories per gram of water at 75°, 115 calories at 300°, and 730 calories at 650° C. After having been heated to 800° C. the adsorbing power of powdered quartz is lost. The adsorption of air, carbon dioxide, etc., is probably negligible compared with that of water. This curve is thought to show the effect of deadsorption of ions from broken bonds. Curves for other anhydrous crystals are similar.

Curve E: The variation of weight with temperature when the energy of association is 100 calories per gram (1,800 cal./mole) through the whole range of temperature from 30° to 800° C. is indicated by curve E. It is computed from the equation

$$\frac{d \, \log \, W}{d(1/T)} {=} \frac{q}{R} \text{ or } \log \, (W/D) {=} \, C/T$$

where C=q/R and log D is an arbitrary integration constant here chosen so that W=1 at  $600^{\circ}$  C. By comparing the slope of another curve with that of the E curve at the same temperature, the energy of association (heat of vaporization) may be estimated.

Curve S: Dried silica hydrogel Si (OH)<sub>n</sub>, obtained by evaporation from repeated solution of silica gel in distilled water (two liters will dissolve nearly a gram at 90° in 24 hours). The dehydration curve is a straight line between 170° and 480° C. At about 300°-C, the water energy is nearly the same as that of water adsorbed on quartz powder, about 100 cal./gram. In the range 50°-100° C., water is more firmly held on the gel than on the quartz, as though a hydrate were attempting to form; in the higher range, 200°-700° C., the curves run nearly parallel. Commercial silica gel and gel freshly precipitated from sodium silicate give curves that are similar but wavy, probably owing to impurities.

Curve A: A typical volcanic ash from west Texas. The amorphous glasses that are the chief constituent of volcanic ash might be expected to give smooth, featureless curves, but few do. Even pyrex glass gives a curve having a decided shoulder. The ash curve shown is the smoothest of many obtained. There is a pronounced

hydrate shoulder at low temperatures, which has disappeared at 160° C. Comparatively low-energy water is driven off at higher temperatures. Most ashes and tuffs show traces of the montmorillonite shoulder at 500° C., owing to its incipient formation on shard surfaces.

Curve M: In strong acids montmorillonite gives up strong bases but essentially no silica; in pure water it gives up silica but only a small proportion of bases. However, in acid solutions of about 0.2 percent concentration bases and silica are removed in the original proportion and may be recovered by evaporation and washed free from soluble salts. No crystalline form is evident in the residue under a microscope. M is the dehydration curve of such a preparation, plotted to half the original scale. Slight shoulders at 490° and 670° C. correspond with those of montmorillonite and saponite.

Curve R: The extraction of the bases from montmorillonite with 20 percent acid leaves an isotropic residue usually over 99 percent silica. It is of interest to compare this residue with silica gel. R is for the

silica residue obtained from a very pure bentonite by twice extracting it for 24 hours with 20 percent hydrochloric acid. The residue contains but 0.15 percent  $Al_2O_3$ . Its dehydration curve is similar to that for pure silica hydrogel (fig. 14, S), but the residual shoulder at 400° C. is still prominent.

Curve D: Diatoms from Ontario, eastern Oregon. This curve is for a very pure white diatomite containing no visible clay material and consisting of only two species of Melosira. The sample used was freed from finer fragments and coarser lumps by washing, settling, decanting, and filtering. Al<sub>2</sub>O<sub>3</sub> amounted to 2.3 porcent, grain density 1.688. The dehydration curve is very similar to that for pure silica hydrogel (fig. 14, S), being straight in the central part and parallel throughout but slightly higher. The high energy of low-temperature water is suggestive of a hydrate.

Curve L: A commercial synthetic magnesium silicate called "Magnesol," extensively used as a bleaching agent for certain kinds of oil. It has a remarkably long straight central portion and no shoulders. It is plotted to half the original scale.

Tempera- ture (°C.)	· Q	E	s	A	М	R	D	L L
50	1. 00544	1. 0540	1. 0560	1. 0408	1. 2020	1. 0832	1. 0825	1. 1690
100	1. 00468	1. 0402	1. 0415	1. 0375	1. 1666	1. 0458	1. 0475	1. 1332
150	1. 00402	1. 0316	1. 0341	1. 0290	1. 1356	1. 0388	1. 0406	1. 1150
200	1. 00345	1. 0251	1. 0297	1. 0161	1. 1100	1. 0333	1. 0356	1. 1024
300	1. 00251	1. 0174	1. 0215	1. 0077	1. 0730	1. 0283	1. 0272	1. 0816
400	1. 00180	1. 0139	1. 0135	1. 0039	1. 0484	1. 0242	1. 0192	1. 0620
500	1. 00120	1. 0117	1. 0058	1. 0020	1. 0260	1. 0122	1. 0110	1. 0420
600	1. 00072	1. 0000	1. 0021	1. 0009	1. 0160	1. 0076	1. 0050	1. 0220
700	1. 00032	. 9982	1. 0006	1. 0003	1. 0080	1. 0045	1. 0021	1. 0116

#### HYDROUS OXIDES AND ZEOLITES

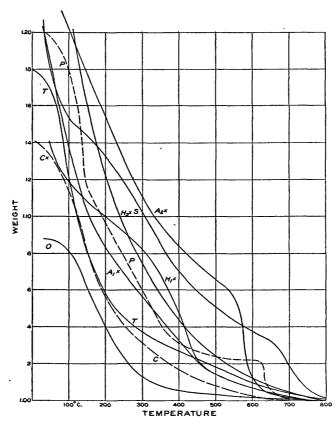


FIGURE 15.—Curves for hydrous oxides and zeolites.

- $A_1$  and  $A_2$ , Allophanes (half scale).
- $H_1$  and  $H_2$ , Aluminum hydroxide (half scale).
- O, Mexican opal.
- S, Sepiolite.
- T, Ptilolite.
- C, Chabazite (half scale).
- P, Phillipsite.

Curves  $A_1$  and  $A_2$ : Allophanes from Kentucky ( $A_2$  from Morehead). Both curves half scale. Both samples are brownish transparent glasses, so dry that fragments rattle. The curve  $A_1$  is devoid of shoulders and probably represents finer-grained material than  $A_2$ , which carries twice as much water and has a shoulder corresponding to saponite. A sample from Liege, Belgium, has the high water of  $A_2$  but only a slight shoulder at 550° C. Despite its high affinity for water, allophane shows no tendency to form hydrates, nor is it at all hygroscopic. An isotropic transparent mineral from Iowa, apparently allophane, was found by thermal analysis to be halloysite, composed of crystals too small to diffract light and lacking definite orientation.

Curves  $H_1$  and  $H_2$ : Aluminum hydroxide. Both curves half scale. Alumina precipitated in the ordinary way from a dilute solution gives a curve with a shoulder near 350° C., like  $H_1$ . By using strong solutions it is possible to secure an amorphous hydrate, represented by  $H_2$ , containing 40 percent water, roomdry, and showing no trace of a shoulder. Curve  $H_2$  was run on such a sample 3 months old, which showed no tendency to revert to gibbsite (fig. 16, G).

Curve O: Mexican opal. From United States I<sup>†</sup>ational Museum collection. The sample was an old cut stone, highly transparent but with a brownish tint. Al<sub>2</sub>O<sub>3</sub> was 0.3 percent. It has a low-temperature shoulder like that of the zeolites (fig. 15, T, C, P) and Texas volcanic ash (fig. 14, A), rather than that of silica gel (fig. 14, S), but with twice as much water. A stable hydrate under room conditions, the hydrate was destroyed at 160° C.

Curve S: Sepiolite, from Asia Minor. From United States National Museum collection. A fine-grained gray claylike material, probably a mixture of hydrous minerals. Sepiolite curves show no definite group characteristics. Individual curves differ considerably from each other and shade off into those of the saponites. Indications are that a pure mineral would give a curve with no shoulders. The two slight shoulders indicate traces of bauxite and saponite.

Curve T: Ptilolite, from Marysvale, Utah. A very pure crystalline sample supplied by W. T. Schaller. Freed from calcite by acid. The curve shows the low-temperature shoulder of the zeolites, analcime, opal and some ashes, but no trace of a montmorillonite,

kaolinite, or saponite shoulder, and is a straight line between 370° and 600° C. Energy of water association is high at both high and low temperatures.

Curve C: Chabazite, from Oregon. From United States National Museum collection. A pure crystal-line material. This is a typical zeolite curve, with low-temperature shoulder, similar to that for ptilolite but higher at low temperatures. Plotted half scale.

Curve P: Phillipsite, from Victoria, Australia. Supplied by United States National Museum. The sample

consists of tiny crystals picked from the inner surface of rock cavities. Characterized by low-temperature shoulders at 20° and 140° C., a straight-line portion between 150° and 350° C., and a pronounced shoulder at 630° C. corresponding to a 1H<sub>2</sub>O hydrate. The 20° C. shoulder is close to 8H<sub>2</sub>O (1.201). A dozen Atlentic and Pacific bottom samples kindly supplied by W. H. Bradley and T. W. Vaughan were tested, but none showed the characteristics of a phillipsite.

Weight ratios for hydrous oxides and zeolites

Tempera- ture (° C.)	$A_1$	A2	$H_1$	$H_2$	0	s	T	C	P
50	1. 365	1. 575	1. 277	1. 530	1. 0871	1. 1790	1. 1686	1. 265	1. 1972
100	1. 268	1. 430	1. 236	1. 422	1. 0813	1. 1535	1. 1200	1. 226	1. 1797
150	1. 203	1. 354	1. 217	1. 321	1. 0615	1. 1424	1. 0795	1. 163	1. 1160
200	1. 168	1. 303	1. 199	1. 244	1. 0393	1. 1332	1. 0582	1. 114	1. 0965
300	1. 103	1. 233	1. 163	1. 150	1. 0130	1. 1030	1. 0374	1. 062	1. 0596
400	1. 0610	1. 167	1. 095	1. 086	1. 0058	1. 0700	1. 0272	1. 0341	1. 0315
500	1. 0242	1. 131	1. 028	1. 047	1. 0039	1. 0507	1. 0183	1. 0164	1. 0240
600	1. 0150	1. 025	1. 015	1. 023	1. 0027	1. 0370	1. 0100	1. 0056	1. 0220
700	1. 0052	1. 003	1. 005	1. 008	1. 0011	1. 0166	1. 0038	1. 0000	1. 0010

#### HYDROUS OXIDES AND RELATED SILICATES

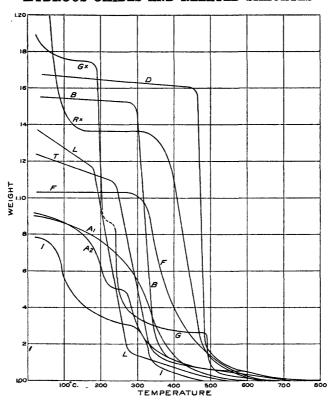


FIGURE 16.-Curves for hydrous oxides and related silicates.

 $A_1$  and  $A_2$ , Analcime. G, Gibbsite (scale, one-third).

D, Diaspore.

L, Limonite. I. Inesite. R, Rectorite ( $\times 2$ ). T, Goethite. B. Brucite. F. Dufrenite.

Curves  $A_1$  and  $A_2$ : Analcime.  $A_1$  represents pure coarsely crystalline material from Wyoming, supplied by W. H. Bradley. It gives a smooth S-shaped curve. Structural water is in the form of a low-temperature hydrate, as with the zeolites.  $A_2$  represents microscopically crystalline material from Arizona, found by C. S. Ross to be altered volcanic ash with a trace of montmorillonite. This curve is roughly parallel to  $A_1$ but has a shoulder at 600° C. where about half the water has escaped, as though a hemi-hydrate were present. Other minerals show a similar difference between coarse-grained and very fine-grained forms.

Curve G: Synthetic gibbsite, Al(OH)<sub>3</sub>, precipitated by the writer from aluminate solution with CO<sub>2</sub>. Scale, one-third. Coarse crystals, very pure. Extremely sharp shoulders at 1H<sub>2</sub>O and 3H<sub>2</sub>O, possibly also at 2H<sub>2</sub>O. Thermostatic control is required to get details between 145° and 240° C. Curves of the higher-grade Arkansas bauxites indicate that they are composed chiefly of gibbsite.

Curve D: Natural diaspore, Al<sub>2</sub>O<sub>3</sub>.H<sub>2</sub>O, from Rolla, Mo., selected by C. S. Ross. The part below 430° C. is a straight line, slightly inclined. The shoulder is sharp and checks well with the second gibbsite shoulder at 480°-490° C.

Curve L, limonite, and curve T, goethite: Limonite, from Avery, N. C.;  $Fe_2O_3$ , 83.4 percent;  $SiO_2$ , 1.4 per-

cent: Al<sub>2</sub>O<sub>3</sub>, 0.6 percent. Goethite, Fe<sub>2</sub>O<sub>3</sub>.H<sub>2</sub>O, Eltered product from marcasite going over to hematite. Both specimens thought to be of high purity. Each curve consists chiefly of straight lines with two sharply defined shoulders. The limonite contains more water below 180° C. but much less than goethite in the range 200° to 300° C. Neither shows any evidence of ferrous iron and neither shows the theoretical amount of hydroxyl at room temperature. Thermal analysis should be a useful research tool in differentiating these minerals.

Curve B: Brucite, from Nevada. White, crystalline, contains traces of R<sub>2</sub>O<sub>3</sub>, CaO, and CO<sub>2</sub>. This curve, also, consists chiefly of two straight lines with a sharp intersection just under 300° C. Below 250° C. it is horizontal and slightly higher than the theoretical curve, owing to the presence of a little magnesite.

Curve I: Inesite, 3CaO.11MnO.15SiO<sub>2</sub>.10H<sub>2</sub>O, from Quinault, Wash. Supplied by W. T. Schaller. The curve consists of two rounded shoulders, one at about 280° C., the other at a low temperature as in the zeolites. Usually classed as a zeolite.

Curve R: Rectorite, from Hot Springs, Ark. Very pure Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>.H<sub>2</sub>O by analysis. Consists of small soft, translucent flakes having high luster. Ordinates are magnified ×2. The shoulder between 300° and 400° C. is similar to those of beidellite and halloysite is horizontal instead of inclined.

Curve F: Dufrenite, from Cherokee County, Ala. the curve for brucite.

but the intermediate part of the curve, 160°-320° C., A hydrous iron phosphate, 2Fe<sub>2</sub>O<sub>3</sub>.P<sub>2</sub>O<sub>5</sub>.3H<sub>2</sub>O. It shows a definite hydrate, stable at 300° C., and resembles

Weight ratios for hydrous oxides and related silicates

Temperature (° C.)	$A_1$	G	D	L	T	В	. I	R	F
50 100 150 200 250	1. 089 1. 086 1. 083 1. 077 1. 069	1. 545 1. 529 1. 525 1. 345 1. 150	1. 167  1. 165	1. 134 1. 128 1. 120 1. 096 1. 040	1. 122 1. 115 1. 112 1. 100	1. 465  1. 460 1. 458	1. 0765 1. 0552 1. 0413 1. 0354 1. 0317	1. 1010 1. 0736 1. 0684 1. 0674 1. 0684	1. 1035 1. 1035 1. 1035 1. 1035 1. 1035
300 350 400 450 500 600	1. 055 1. 031 1. 014 1. 006 1. 003 1. 001	1. 105 1. 090 1. 084 1, 079 1. 027 1. 003	1. 163 1. 162 1. 160 1. 015 1. 005	1. 014 1. 008 1. 004 1. 002	1. 050 1. 015 1. 010	1. 430 1. 080 1. 030	1. 0275 1. 0080 1. 0042 1. 0038 1. 0030 1. 0019	1. 0684 1. 0660 1. 0544 1. 0250 1. 0064 1. 0002	1. 1010 1. 0712 1. 0408 1. 0245 1. 0142 1. 0033

#### KAOLIN GROUP

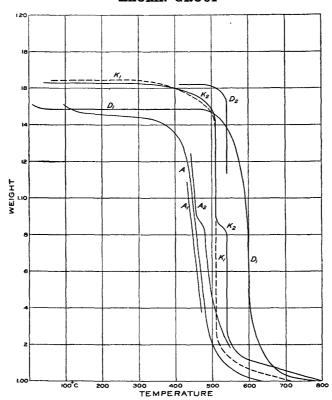


FIGURE 17.-Curves for the kaolin group.

A,  $A_1$ , and  $A_2$ , Anauxite.  $K_1$  and  $K_2$ , Kaolinite.  $D_1$  and  $D_2$ , Dickite.

The minerals of the kaolin group—anauxite (?), kaolinite, dickite, and nacrite—have been classified and critically discussed by Ross and Kerr.¹ On the evidence of their thermal analysis curves alone, the anauxites form a group quite distinct from the kaolinites. The nacrite curves differ widely from each other and cross those of both dickite and kaolinite. The data on the kaolin groups used in this report, except those for the kaolinites from Bishop, Calif.  $(K_2)$ , Stillwater, Mont., and Gordon, Ga., and for the dickite from Pennsylvania  $(D_1)$ , are taken from 1927 determinations by J. G. Fairchild.

Curves A,  $A_1$ , and  $A_2$ : Anauxite group.  $A_1$  refers to a sample from the Mokelumne River, Calif. It has a considerable range (0.1 to 3 mm.) of grain sizes and shows no indication of a shoulder near the half-weight ratio (1.08). The intermediate curve, A, fully drawn, is for a sample from the Newman pit near Ione, Calif., with grain sizes from 1.5 mm. down. Curve  $A_2$  is for a fine-grained (0.002 to 0.02 mm.) sample from Franklin, N. C. These anauxite curves have a decided slope, whereas the kaolinite curves drop vertically. They break at a temperature  $50^{\circ}$  C. lower than the kaolinite curves. In the lower range of temperature  $(100^{\circ}-350^{\circ}$  C.), all anauxite curves available exhibit a definite slope and are lower than they should be

theoretically (2H<sub>2</sub>O=1.1628), whereas the kaolinite curves are full height and horizontal in this region.

Curves  $K_1$  and  $K_2$ : Typical kaolinites.  $K_1$  is from Ione, Calif.,  $K_2$  from Bishop, Calif. The curves show horizontal tops close to the theoretical 1.162, and the parts between 485° and 540° C. (chiefly 500°-510° C.) are vertical. The finer-grained materials (colloids?) show a small shoulder halfway down. On thermal evidence alone, the Mexican dickite,  $D_2$ , might be a pure kaolinite. Curves for other samples from Pontiac, S. C., Jerome, Ariz., Stillwater Range, Mont., and Gordon, Ga., all fall within the range shown by  $K_1$  and  $K_2$ .

Curves  $D_1$  and  $D_2$ : Dickite.  $D_1$  refers to material from Pennsylvania. The large lustrous platy crystals (0.3 mm.) are very pure and without a visible trace of foreign substances. The curve is horizontal as far as 580° C. The vertical part is at 600° C. The low-temperature part indicates a 1.1482 weight ratio corresponding to 1.83 H<sub>2</sub>O instead of 2H<sub>2</sub>O. The toe of the curve is horizontal at 800° C., indicating that the base weight is correct.  $D_2$  refers to a clay from Cusihuiriachic, Mexico (crystals 0.03 mm.). The curve is horizontal (1.162) as far as 480° C., then drops straight down at 540°.

Nacrite was available only in small sample from Brand, Saxony, and from Pikes Peak, Colo. The thermal curves of these differ widely. Each shows

<sup>&</sup>lt;sup>1</sup> Ross, C. S., and Kerr, P. F., The kaolin minerals: U. S. Geol. Survey Prof. Paper 165, pp. 151-180, 1931,

two shoulders: the material from Brand at 450° and A Russian sample, said to be nacrite, has the thermal 620° C.; that from Pikes Peak at 550° and 650° C. | curve of anauxite.

Weight ratios for the kaolin group

		Anauxite				Kaolinite	-		Dickite	Na	crite
Temperature (°C)	$A_1$	A	A2	$K_1$	K2	Pontiac, N. C.	Jerome, Ariz.	Stillwater, Mont.	$D_1$	Brand, Saxony	Pikes Frak, Cole.
100 200 300 400 450	1. 140 1. 137 1. 134 1. 127 1. 078	1. 150 1. 146 1. 144 1. 136 1. 096	1. 163 1. 163 1. 162 1. 153 1. 105	1. 164 1. 164 1. 164 1. 160 1. 155	1. 163 1. 163 1. 163 1. 160 1. 157	1. 169 1. 168 1. 167 1. 062 1. 056	1. 165 1. 165 1. 165 1. 161 1. 159	1. 161 1. 160 1. 159 1. 158 1. 157	1. 149 1. 149 1. 149 1. 149 1. 149	1. 173 1. 173 1. 167 1. 167 1. 163	1. 149 1. 149 1. 149 1. 147 1. 146
475 500 525 550 575	1. 033 1. 012 1. 008 1. 005 1. 003	1. 050 1. 020 1. 012 1. 008 1. 005	1. 083 1. 046 1. 029 1. 019 1. 014	1. 152 1. 147 1. 018 1. 013 1. 009	1. 154 1. 149 1. 085 1. 021 1. 014	1. 051 1. 041 1. 020 1. 016 1. 014	1. 156 1. 087 { 1. 076 1. 032 1. 023 1. 019	1. 156 { 1. 152 1. 086 } 1. 052 1. 031 1. 021	1. 148 } 1. 147 1. 143 1. 135 1. 119	1. 157 1. 134 1. 096 1. 075 1. 062	1. 145 1. 144 1. 143 { 1. 138 1. 106 1. 075
600 650 700	1. 001 1. 000 1. 000	1. 003 1. 000 1. 000	1. 011 1. 007 1. 002	1. 007 1. 004 1. 001	· 1. 012 1. 009 1. 006	1. 012 1. 009 1. 006	1. 015 1. 009 1. 004	1. 015 1. 006 1. 002	1. 070 1. 012 1. 003	1. 050 1. 016 1. 009	1. 066 { 1. 057 1. 028 1. 007

#### MONTMORILLONITES

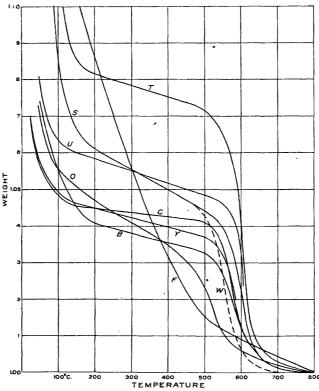


FIGURE 18.-Curves for montmorillonites.

- S, Pure bentonitic montmorillonite.
- W, Bentonitic montmorillonite, slightly weathered.
- C, High-potash bentonite from Suifu, China.
- B, High-potash bentonite from High Bridge, Ky.
- Y, High-sodium bentonite from Upton, Wyo.
- U, High-sodium bentonite from Upton, Utah.
- T, Bentonite from Whitewright, Tex.
- O, Fuller's earth from Olmstead, Ill.
- F, Fuller's earth from Florida.

The curves shown in figure 18 represent investigations of pure or nearly pure montmorillonite as determined by petrographic and X-ray analysis. Water and alkalies and alumina-silica ratios vary considerably. The various members of the montmorillonite group are considered in another section, represented on figure 19.

Thermal curves indicate several classes of montmorillonites: (1) Pure normal fresh bentonite, collected well below the zone of weathering, and modifications resulting from weathering and storage in dry and in saturated atmospheres; (2) bentonites high in alkalies, whose curves have a less inclined central part than those of group (1); and (3) very waxy hygroscopic bentonites, much higher in water than normal, as though in colloidal condition. All these curves are characterized by (a) a very steep initial part from room temperature to about 160° C., (b) a uniform slope in the middle range, 200°-450° C., and (c) a final steep drop in the region  $500^{\circ}-600^{\circ}$  C. The beidellites (fig. 19, B, R, G) drop at lower temperatures (400°-500° C.) and the saponites (fig. 19, T) at higher temperatures  $(630^{\circ}-680^{\circ})$ 

The montmorillonite clays are used industrially for bleaching oils and fats, for bonding molding sands, for stiffening drilling muds, and in many other ways. Five types and several subtypes are clearly recognized. These types are (1) sodium-saturated swelling bentonites of the Wyoming class, useful for their gelling and detergent properties but useless for decolorizing, (2) pure normal bentonites capable of high activation as bleaching agents but inactive until acid treated, (3) clays that are active untreated (like the fuller's earths) and also capable of high activation, (4) clays such as the Porters Creek, which are active urtreated and indifferent to acid treatment, and (5) the normal fuller's earths from Georgia and Florida, which are considerably deactivated by acid treatment. Only the high-sodium clays are in class 1, whereas the highpotash bentonites are in class 3. The property of bonding molding sands is dependent upon hygroscopicity, and selected deposits of clay of all five classes are used for the purpose. All classes except (1) are used for bleaching oil.

Curve S: Normal pure bentonitic montmorillonite. This standard curve is identical with that for montmorillonite from the Vicksburg (of Oligocene age) at Polkville, Miss. It is very near those for clays from Mainburg, Bavaria; Tatatila, Mexico; Rosedale, Alberta; Chito, Ariz.; Mojave and Otay, Calif.; Bogalusa, La.; and many others. Aside from the three general characteristics mentioned above, the curve for a pure normal bentonite may be quicky identified by the value 1.05 (1H<sub>2</sub>O) at  $400^{\circ}$  C. and by its slope in a straight line from 1.061 at 200° to 1.048 at 520° C. When plotted as log (water) versus 1/T, this curve breaks up into three straight lines, the slopes of which— $d \log W/d$  (1/T)—indicate energies of association of 173 calories per gram from 30° to 150° C., 37.6 from 150° to 500° C., and 1,068 from 500° to 650° C., with a fairly sharp transition (less than 50° C.). The loss in weight at high temperatures is due to loss of hydrogen and hydroxyl in the crystal lattice; that at intermediate temperatures represents the escape of water adsorbed on or between lattice planes; and that at low temperatures represents the escape of water that is present as simple moisture, although its energy of association is four times that of the adsorbed water.

Curve W (dashed): Bentonitic montmorillonite, slightly weathered. This curve represents bentonite taken from a place so near the surface that atmospheric agencies have weakened its lattice structure. Bentonite stored for a few months at room temperature, either in a dry or in a water-saturated atmosphere, shows a similar alteration of curve S toward W. Anhydrous impurities (quartz, feldspar), of course, lower the whole curve, in proportion to their quantity, to the position of curve B or lower. Many montmorillonite curves show the results of impurities and weathering.

Curves C, B, Y, and U: Bentonite. C, from Suifu, China; high potash (5.16 percent); known as "soap clay." B, from High Bridge, Ky.; of Ordovician age; high potash (5.72 percent); contains feldspar. Y, from Upton, Wyo., sodium saturated. U, from Upton, Utah; Na<sub>2</sub>O, 2.75 percent; low slope, high breaking temperature. These curves are all of decreased slope compared with that for normal bentonite, indicating that the interlattice adsorbed water is much less firmly held when considerable alkali is present. If sodium causes an effect different from potassium, these curves do not show it. These curves are normal in their high-temperature shoulders.

Curve 7: Bentonite from the Austin chalk (of Upper Cretaceous age), near Whitewright, Tex. Contains many biotite grains; very waxy; hygroscopic like a dried colloid. Although normal in appearance, texture, fracture, etc., it suggests a bentonite abnormally high in water. Similar elevated curves are common for the beidellites and for other clays whose curves break around 450° C., but they are rare in the montmorillonites. Although high in water retention, this clay also holds its water firmly, as shown by the 600° C. break.

Curves O and F: Fuller's earth. O is from Porters Creek clay (of Paleocene age), Olmstead, Ill.; nearly normal montmorillonite (curve is lower and breaks at 500° instead of 580° C., indicating that material is slightly weathered and impure). Porters Creek clay from Pinson Hill, Tenn., gives curves like O. F is from the Hawthorn (of middle Miocene age) of Florida; slight humps at 190° and 310° C. are not shown in figure 18. Although curve F shows no shoulders, most curves of this type show near 200° and 300° C. minor shoulders that probably indicate hydrates that become unstable at those temperatures. Clay from Attabulgus, Ga., gives curves with humps at 150° and 310° C., similar to F. Some samples from the same pit as O, Olmstead, Ill., gave curves like F, having no hightemperature shoulder but one or two shoulders at lower temperatures. The fuller's earths, whether from the Porters Creek clay of Tennessee and Illinois or from the Hawthorn of Georgia and Florida, all exhibit two widely different types of thermal curves, even in samples from the same or neighboring pits. One type, O, evidently represents nearly normal montmorillonite; the other type, F, shows no lattice shoulder and resembles the featureless curves of an amorphous hydrogel. The thermal evidence is that either two different minerals, montmorillonite and "attapulgite" (?) are present or that the same substance occurs in two widely different forms, one a hydrogel.

Weight ratios for montmorillonites

Tempera- ture (° C.)	s	С	В	Y	T	U	o	F
100	1. 0860	1. 0483	1. 0552	1. 0492	1. 1090	1. 0635	1. 0555	1. 1290
200	1. 0610	1. 0449	1. 0408	1. 0449	1. 0815	1. 0585	1. 0470	1. 0870
300	1. 0555	1. 0437	1. 0380	1. 0424	1. 0784	1. 0551	1. 0408	1. 0555
400	1. 0500	1. 0426	1. 0356	1. 0398	1. 0753	1. 0518	1. 0348	1. 0316
500	1. 0444	1. 0411	1. 0326	1. 0372	1. 0715	1. 0484	1. 0234	1. 0154
550	1. 0395	1. 0340	1. 0243	1. 0320	1. 0650	1. 0462	1. 0108	1. 0118
600	1. 0225	1. 0122	1. 0122	1. 0130	1. 0400	1. 0316	1. 0058	1. 0092
700	1. 0014	1. 0015	1. 0020	1. 0013	1. 0030	1. 0020	1. 0020	1. 0045

#### MONTMORILLONITE GROUP

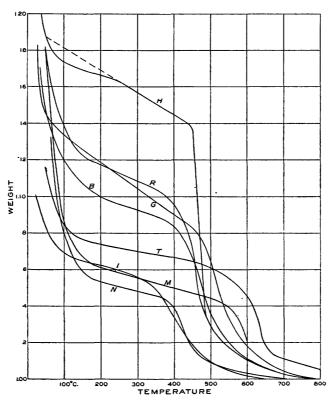


FIGURE 19.—Curves for the montmorillonite group.

- M, Standard curve for normal montmorillonite (see also fig. 18, S).
- B, Beidellite.
- G. Thin bed of dried gel (beidellite?).
- R, "Racewinite."
- N, Nontronite.
- H, Halloysite.
- I, Illite.
- T, Saponite.

In figure 19 are assembled typical dehydration curves for the various members of the montmorillonite group including beidellite, nontronite, and saponite, and the related halloysite and illite, as well as montmorillonite itself. The type material from Montmorillon, France, does not give the curve for normal bentonitic clay but a curve that has two shoulders, at about 480° and 640° C., one or both of which appear to represent minerals other than that generally accepted as montmorillonite. Pyrophyllite, Al<sub>2</sub>O<sub>3</sub>.4SiO<sub>2</sub>.H<sub>2</sub>O (fig. 21, P), has a constant weight of 1.05 up to 500° C., then follows the normal montmorillonite shoulder and toe very closely.

Curves B, R, and G: Beidellite. B, a very pure clay mineral replacing calcareous shells and excluding all calcium, from Pontotoc, Miss. R, "racewinite," from a seam in a Utah copper deposit, described by Winchell; 2 very close to beidellite in composition but optically positive. G, Sample from thin bed of dried colloidal material apparently intermediate between beidellite and montmorillonite; from West Chester, Pa. At 400° C. water is slightly higher than normal in B, and considerably higher in R. These three curves are

typical of many beidellites and well show the range of variation found in a dozen clays of this class studied by the writer.

Curve N: Nontronite, from Santa Rita, N. Mex. This curve is typical of three nontronites studied. Samples from Sandy Ridge, N. C.; Spokane, Wash.; Warthen, Ga.; and others give nearly identical curves. These curves differ little from those for weathered beidellites. Water appears low chiefly on account of the heavier iron constituent.

Curve H: Halloysite, from Liege, Belgium. Curves of samples from Adams County, Ohio; Rome, Ga.; and Anamosa, Iowa, are almost identical with the type material plotted in H. All halloysite curves are characterized by their height (1.145 at 400° C.), by their straight line slope between 250° and 450° C., and by their dip in the range between 50° and 250° C. The steep drop occurs between 450° and 500° C. There has been some discussion of a change in halloysite near 50° C., but according to these curves such a change should be sought at 250° C. Several glauconite curves (fig. 20, G) also show a similar dip followed by a straight line but at 1.05 instead of 1.15 in weight ratio.

Curve I: Illite. The curve is plotted from data

<sup>&</sup>lt;sup>9</sup> Winchell, A. N., Racewinite, a peculiar mineral from ore deposits in Utah: Econ. Geology, vol. 13, pp. 611-613, 1918.

obtained by R. E. Grim <sup>3</sup> in 1937 on selected material and is given here to show its resemblance to that of nontronite. It differs widely from the thermal curves for such minerals as sericite and muscovite.

Curve T: Saponite, from Hector, Calif. This white swelling clay is normal saponite except for small

Grim, R. E., personal communication.

amounts of Na and Li. The dried hydrogel gave a curve having shoulders at 500°, 600°, and 960° C. and was still losing weight at 1000° C., the limit of the apparatus. The curve is tentative and is the smoothed mean of five curves, which differ considerably in detail and are uncertain as to base weight. Three minor shoulders are omitted.

Weight ratios for the montmorillonite group

Temperature	M	В	G	R	N	Н	I	T
50 100 150 200 300	1. 182 1. 086 1. 065 1. 061 1. 055	1. 148 1. 120 1. 107 1. 100 1. 093	1. 145 1. 134 1. 126 1. 119 1. 105	1. 182 1. 139 1. 123 1. 117 1. 109	1. 186 1. 080 1. 059 1. 053 1. 048	1. 188 1. 174 1. 169 1. 166 1. 157	1. 083 1. 069 1. 065 1. 062 1. 056	1. 113 1. 085 1. 077 1. 074 1. 070
350 400 450 500 550 600 700	1. 033 1. 050 1. 047 1. 044 1. 039 1. 020 1. 001	1. 089 1. 084 1. 067 1. 036 1. 020 1. 011 1. 003	1. 097 1. 090 1. 083 1. 064 1. 034 1. 018 1. 004	1. 104 1. 096 1. 072 1. 029 1. 017 1. 011 1. 003	1. 046 1. 039 1. 017 1. 009 1. 006 1. 003 1. 000	1. 151 1. 145 1. 138 1. 029 1. 017 1. 011 1. 003	1. 050 1. 034 1. 019 1. 010 1. 005 1. 002 1. 000	1. 069 1. 067 1. 065 1. 061 1. 055 1. 045 1. 011

#### CHLORITES AND RELATED SILICATES

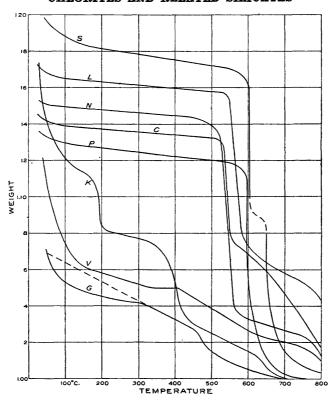


FIGURE 20.—Curves for chlorites and related silicates.

- S, Serpentine.
  P. Picrolite.
  - ine. e.
- C, Chlorite.
  L. Clinochlore.
- N, Penninite.
- K, Paligorskite.
- V, Vermiculite.
  G. Glauconite.
- This group comprises mainly chlorites and related hydrous magnesium alumino-silicates. They have not been studied sufficiently to permit many general conclusions to be drawn, but the nature of the dehydration curves to be expected in further work is indicated.

Curve S: Fibrous serpentine, supplied by W. T. Schaller. Very pure, Fe<sub>2</sub>O<sub>3</sub> but 0.3 percent. The curve of this sample shows the characteristic rather sharp shoulder and steep drop between 600° and 650° C. No points were obtained within that range but the alinement of the branches suggests a shoulder half way down. Granular serpentines containing nickel and 5 to 7 percent of FeO show a lower curve having a greater slope below 500° C. and a more rounded shoulder but a break at the same temperature.

Curves L, N, C, and P are similar to that for serpentine (S), but all except picrolite have a second shoulder at a higher temperature. C, chlorite, from Danville, Va.; base weight at 930°, 1.006 at 830° C. L, Clinochlore, from Chester County, Pa.; approximately 5MgO.Al<sub>2</sub>O<sub>3</sub>.3SiO<sub>2</sub>.4H<sub>2</sub>O. N, Penninite, from Paradise Range, Nev.; content of (Na, K)<sub>2</sub>O less than 0.1 percent; base weight at 870° C. P, Picrolite, from Baltimore County, Md.; dark-green fibrous serpentine.

Curve K: Paligorskite ("Mountain leather"), from Montana. A gray fibrous, extremely tough spec men. The curve has three shoulders, at 170°, 340°, and 620° C., with intervening straight-line parts.

Curve V: Vermiculite, from North Carolina. The base weight (anhydrous) is uncertain and has been arbitrarily chosen as 1.050 at 340°-405° C. Al' free moisture has been driven off below 340° C. The characteristic swelling occurs above 400° C. Curves for vermiculites from other localities are similar in general character but differ in detail.

Curve G: Glauconite, from Lyons Wharf, Md. Light-green, opaque "greensand." Of the four glauconites studied—from Huntington, Oreg., Bonne Terre, Mo., Sewell, N. J., and Lyons Wharf, Md.—the Fonne Terre sample contained the most water, 1.051 at 400° C. On account of admixed quartz grains, the sample from Lyons Wharf contained the least water, but it shows most clearly the characteristic shape of the curve, which is concave from 50° to 320° C. and a straight line approaching the shoulder at 470° C. A correction of only 1.016 in base weight would bring the highest and lowest curves together at 400° C.

Weight ratios for chlorites and related silicates

Tempera- ture (° C.)		L	N	C	P	K	v·	G
50	1. 196	1. 168	1. 151	1. 142	1. 133	1. 141	1. 107	1. 068
100	1. 188	1. 165	1. 150	1. 139	1. 130	1. 121	1. 074	1. 053
150	1. 184	1. 164	1. 149	1. 138	1. 128	1. 114	1. 062	1. 048
200	1. 181	1. 163	1. 148	1. 137	1. 127	1. 085	1. 058	1. 045
.300	1. 178	1. 161	1. 146	1. 136	1. 125	1. 077	1. 052	1. 042
400	1. 175	1. 159	1. 145	1. 134	1. 122	1. 054	1. 050	1, 033
500	1. 172	1. 158	1. 140	1. 132	1. 120	1. 025	1. 039	1, 015
550	1. 170	1. 152	1. 066	1. 080	1. 119	1. 020	1. 033	1. 009
600	1. 161	1. 074	1. 033	1. 069	1. 060	1. 015	1. 027	1. 005
650	1. 080	1. 064	1. 030	1. 059	1. 012	1. 006	1. 022	1. 002
700	1. 015	1. 058	1. 027	1. 047	1. 003	1. 001	1. 020	1. 000
800	1. 000	1. 043	1. 013	1. 017	1. 000	1. 000	1. 010	1. 000



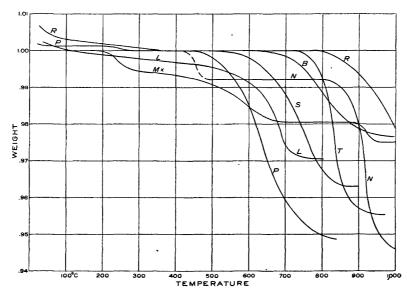


FIGURE 21 -- Curves for micas

P, Pyrophyllite.L, Lepidolite.N, Taeniolite.T. Talc.

R, Tremolite asbestos. S, Sericite.
M, Biotite (X3).
B. Beryl.

Figure 21 shows dehydration curves of miscellaneous minerals, chiefly micas, in which there is a small but characteristic weight decrease, usually at a high temperature. Several specimens of sericite and of pyrophyllite but only single specimens of other minerals were investigated. These minerals hold no water as such under room conditions but all weight losses appear to be caused by the driving out of H and OH from the crystal lattice; hence base weights are chosen at lower temperatures as representative of the mineral. All weights are corrected for air density.

Curve P: Pyrophyllite, Al<sub>2</sub>O<sub>3</sub>.4SiO<sub>2</sub>.H<sub>2</sub>O, transparent radial crystals of a rosette that contained no more than traces of impurities. Swells like vermiculite on heating above 500° C. The material mined at Hemp, N. C., contains some potash, probably in sericite. Pyrophyllite mined near Staley, N. C., is partly in rosette form and very pure. Base weight between 300° and 400° C. The shoulder and toe of this curve are closely similar to those for pure montmorillonite, as would be expected in two minerals having the same internal lattice structure.

Curve L: Lepidolite, from Manitoba. The 3 percent water in this sample is lost very gradually, the structure breaking down only at 690° C. As none of the curve is horizontal, the base weight was taken at the usual 110° C.

Curve N: Taeniolite, from Magnet Cove, Ark. Collected by H. D. Miser. Pure KLiMg<sub>2</sub>Si<sub>4</sub>O<sub>10</sub>F<sub>2</sub>, as determined by R. E. Stevens, in 0.5 mm. crystals. Base weight between 100° and 425° C. The weight is constant up to 420° C.; between 420° and 490° C., 0.80 percent of water is driven off, and the anhydrous

mineral is stable up to 810° C., above which the fluorine is gradually driven off. At the last observed point (1110° C.), the fluorine was not completely removed. An ignition loss for water should not be determined above 800° C., and preferably not above 600° C.

Curve T: Talc. Transparent, colorless.  $R_2O_3$ , 0.36 percent; Fe and Ca absent. The curve shows no weight loss as far as 730° C., and then a rather steep drop which becomes complete at 930° C.

Curve R: Tremolite asbestos, from near Baltimore, Md. This sample contained 0.3 percent water at 100° C. This was lost at a uniform rate up to 350° C. The weight remained constant in the range 350° to 780° C., then fell off in a rounded shoulder. The last observation was at 990° C.

Curve S: Sericite, from near Prince Rupert, British Columbia. Lustrous massive material. Its loss of weight, beginning at about 500° C. amounted to 3.7 percent. Of the other two samples tested, that from Franklin, N. C., lost 3.0 percent, and that from near Baltimore 4.2 percent, with the break occurring between 500° and 550° C.

Curve M: Biotite, from near Philadelphia, Pa. The ordinates have been increased three times to show better the three shoulders at about 200°, 500°, and 900° C., with weight losses of 0.2, 0.5, and 0.2 percent, indicating different associations of hydroxyl and/or fluorine in the structure.

Curve B: Beryl, from Warren Depot, Me. Weight constant up to 630° C., followed by a rounded shoulder extending above 800° C. Constant weight is approached very slowly even at 950° C.

Phlogopite showed no change in weight up to 1025° C.

Weight ratios for micas

Tempera- ture (° C.)	P	L	N	T	. R	s	<i>M</i>	В
100 200 300 400 500-	1. 0014 1. 0014 1. 0000 1. 0000 . 9980	1. 0000 . 9989 . 9978 . 9969 . 9955	1. 0000 1. 0000 1. 0000 1. 0000 . 9921	1. 0000 1. 0000 1. 0000 1. 0000 1. 0000	1. 0030 1. 0018 1. 0007 1. 0000 1. 0000	1. 0000 1. 0000 1. 0000 1. 0000 1. 0000	1. 0000 . 9998 . 9945 . 9935 . 9908	1. 0000 1. 0000 1. 0000 1. 0000 1. 0000
600 700 800 900 1000	. 9850 . 9595 . 9496 . 9486 . 9486	. 9912 . 9739 . 9705 . 9705	. 9921 . 9921 . 9921 . 9800 . 9460	1. 0000 1. 0000 . 9925 . 9572 . 9552	1. 0000 1. 0000 . 9997 . 9936 . 9788	. 9980 . 9880 . 9675 . 9630 . 9630	. 9846 . 9805 . 9805 . 9800 . 9750	1. 0000 . 9983 . 9885 . 9787 . 9765

#### CARBONATES

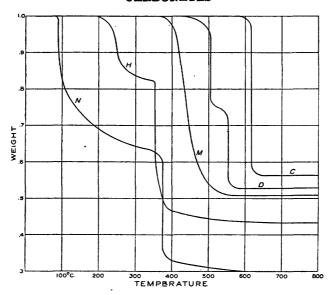


FIGURE 22.-Curves for carbonates.

C, Calcite.D, Dolomite.M, Magnesite.

H, Hydromagnesite.
N. Nesquehonite.

The curves of figure 22 illustrate the behavior of certain carbonates and hydrocarbonates on heating. The thermal decomposition of the carbonates is an extremely slow and steady process, particularly at temperatures just above the first break; however, the attainment of a definite weight with CO<sub>2</sub> only partly removed is beyond question. Diffusion of CO<sub>2</sub> out of the crystal fragments limits the rate of approach to this constant weight. With crystals ground to 150 mesh (0.1 mm.), the rate for calcite is about 1 mg. in 24 hours per gram of samples.

Curve C: Calcite, a perfect crystal; CaO, 56.88 percent. Ground to 0.1 mm. fragments. The main break is between 615° and 620° C. with a tailing off of less than 30° C. The point at 618° C. required 44 days to reach constant weight; at 642°, 31 days. Coarsely crystalline marble gave an identical curve. The aragonite form of CaCO<sub>3</sub> has not yet been investigated by the writer.

Curve D: Dolomite, from Cockeysville, Md. Extracted with 2 percent HCl by R. C. Wells. A double break at 505° and 553° C. is indicated by all samples.

The details of the transition between these two drops remains to be worked out. Another crystalline sample selected by C. S. Ross broke at 515° and 565°. Another from Joplin, Mo., gave a curve like a mixture of calcite and magnesite.

Curve M: Magnesite, from Cote St. Paul, Quebec. Optically pure crystal; traces of calcium and iron, no silica. The first break is at about 420° C. Between 450° and 600° C. the curve is uncertain, as the three runs made did not agree.

Curve H: Hydromagnesite, 3MgCO<sub>3</sub>.Mg(OH)<sub>2</sub>. 3H<sub>2</sub>O. Large natural crystals from Luna, N. Mex., supplied by Eugene Callaghan. The curve shows two shoulders at 220° and 355° C., apparently corresponding to H<sub>2</sub>O and CO<sub>2</sub> alone, but gas tests by R. C. Wells indicate that the gases are evolved as a mixture through the intermediate range. The runs agree very well. Two runs took 9 months.

Curve N: Nesquehonite, MgCO<sub>3</sub>.3H<sub>2</sub>O; pure, crystalline. Synthetic, prepared by R. C. Wells. Shows shoulders at 90° and 375° C.

Weight ratios for carbonates

Tempera- ture (° C.)	C	D	М	Н	N
50 100 150 200	1. 000 1. 000 1. 000 1. 000	1. 000 1. 000 1. 000 1. 000	1. 000 1. 000 1. 000 1. 000	1. 000 1. 000 1. 000 . 998	1. 000 . 825 . 736 . 691
300 400 500 600	1. 000 1. 000 1. 000 1. 000 . 995	1. 000 1. 000 1. 000 . 956 . 528	1. 000 1. 000 . 975 . 540 . 508	. 900 . 837 . 467 . 445 . 435	. 661 . 643 . 330 . 310 . 300

## INDEX

	Page
Abstract	197
Allophane	202
Aluminum hydroxide	202 204
Analcite. See Analcime.	201
Anauxite	206
Anhydrous crystalline minerals	200
Ash, volcanic 200	)-201
"Attapulgite," possible occurrence of	209
Austin chalk, bentonite from 208	3, 209
Base weight of minerals, temperature selected for	
Bauxite	204
Beidellite	210
Bentonite, curves of	
silica residue from 200	
Beryl	214
Biotite	214
Brucite	204
0.1 %	014
Carbonates	216 216
Chabazite 20i	
Chlorite	212
Clay from	
Adams County, Ohio	210
Anamosa, Iowa	210
Attapulgus, Ga	209
Bishop, Calif	206
Bogalusa, La.  Brand, Saxony 206	208
Chito, Ariz	208
Cusihuiriachic, Mexico	206
Florida208	
Franklin, N. C.	206
Gordon, Ga	206
Hector, Calif210	
High Bridge, Ky	
lone, Calif	206
Jerome, Ariz Liege, Belgium	206 210
Mainburg, Bavaria.	208
Mojave, Calif.	208
Mokelumne River, Calif	206
Montmorillon, France	210
Omstead, Ill	, 209
Otay, Calif	208
Pennsylvania	206
Pikes Peak, Colo	⊢207 209
Polkville, Miss	208
Pontiac, S. C.	206
Pontotoc, Miss	210
Rome, Ga	210
Rosedale, Alberta	208
Russia	207
Sandy Ridge, N. C.	210
Santa Rita, N. MexSpokane, Wash	210 210
Stillwater Range, Mont	206
Suifu, China 208	
Tatatila, Mexico	208
Upton, Utah 208	
Upton, Wyo 208	
Warthen, Ga	210
West Chester, Pa	210
Wintewright, 1 ex	
Clinochlore	212
Dehydration, procedure in 197	-198
Diaspore	204
Diatomite 200	
Dickite	206

	P	age
Dolomite	-	216
Dufrenite	204,	205
Energy of association with water, constant	-	200
Feldspar		200
Fuller's earth	208,	209
Gibbsite		204
Glass		200
Glauconite		204
Goethite	-	204
Halloysite		210
Hawthorn formation, fuller's earth from	202	
Hydromagnesite, curve of	<b>~</b> 00,	216
Hydrous oxides	202	
djulous onidos		
Illite, curve of	210-	-211
Inesite, curve of		204
Kaolinite, curves of	_	206
Laboratory methods and equipment	177-	198
Lepidolite		214
Limonite	-	204
Magnesite		216
Magnesium silicate, synthetic	200,	201
'Magnesol''	200,	
Marble		216
Micas		
residue from 2		
	, · · · · ,	201
Mountain laathar See Paligarchita		
Mountain leather. See Paligorskite.		
	206-	-207
Nacrite		-207 216
	_	
Nacrite Nesquehonite Nontronite	-	216
Nacrite Nesquehonite Nontronite	- -	216 210 202
Nacrite Nesquehonite Nontronite	- -	216 210 202
Nacrite	- - 202-	216 210 202 -205
Nacrite	- - 202-	216 210 202 -205 212
Nacrite	- - 202- - -	216 210 202 -205 212 212
Nacrite	- 202- - - - 202,	216 210 202 -205 212 212 203
Nacrite	- 202- - - 202,	216 210 202 -205 212 212 203 214
Nacrite Nesquehonite Nontronite  Dpal  Dxides, hydrous  Paligorskite  Penninite  Philipsite  Philogopite  Picrolite	- 202- - - 202,	216 210 202 -205 212 212 203 214 212
Nacrite Nesquehonite Nontronite  Dal Dxides, hydrous  Paligorskite Penninite Philipsite Phogopite Picrolite Porters Creek clay, fuller's earth from	- 202- - - 202, - - 208,	216 210 202 -205 212 212 203 214 212 209
Nacrite	- - 202- - - 202, - - 208, 202-	216 210 202 -205 212 212 203 214 212 209 -203
Nacrite Nesquehonite Nontronite  Dpal Dxides, hydrous Paligorskite Penninite Philipsite Philogopite Picrolite Orders Creek clay, fuller's earth from Pripolitie	- - 202- - - 202, - - 208, 202- 210,	216 210 202 -205 212 212 203 214 212 209 -203
Nacrite Nesquehonite Nontronite  Dpal Dxides, hydrous Paligorskite Penninite Philipsite Philogopite Picrolite Orders Creek clay, fuller's earth from Pripolitie	- - 202- - - 202, - - 208, 202- 210,	216 210 202 -205 212 212 203 214 212 209 -203
Nacrite	- - 202- - - 202, - - 208, 202- 210,	216 210 202 -205 212 212 203 214 212 209 -203 214
Nacrite Nesquehonite Nontronite  Dpal Dxides, hydrous Paligorskite Penninite Philipsite Philogopite Picrolite Orders Creek clay, fuller's earth from Pripolitie	- - 202- - - 202, - - 208, 202- 210,	216 210 202 -205 212 212 203 214 212 209 -203 214
Nacrite Nesquehonite Nontronite  Dpal Dxides, hydrous  Paligorskite Penminite Phillipsite Philogopite Picrolite Porters Creek clay, fuller's earth from  Ptilolite Pyrophyllite  Quartz, powdered	- - 202- - - 202, - - 208, 202- 210,	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210
Nacrite Nesquehonite Nontronite  Dpal Dxides, hydrous Paligorskite Penninite Philipsite Phologopite Picrolite Porters Creek clay, fuller's earth from Ptilolite Pyrophyllite Quartz, powdered  'Racewinite'' Rectorite		216 210 202 205 212 212 203 214 212 209 -203 214 200 210 -205
Nacrite	- - 202- - - 202, - - 208, 202- 210, -	216 210 202 205 212 212 203 214 212 209 -203 214 200 210 2205
Nacrite Nesquehonite Nontronite  Pall	- - 202- - - 202, - - 208, 202- 210, - - 204-	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 -205
Nacrite	- - 202- - - 202, - - 208, 202- 210, - - 204-	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 205 211 202 214
Nacrite Nesquehonite Nontronite  Popal  Patigorskite Penninite Philipsite Philipsite Picrolite Porters Creek clay, fuller's earth from Pitilolite Pyrophyllite Pyrophyllite Rectorite Saponite Sepolite Sepolite Sepolite Sepolite Sepolite Seponite Sepolite Seponite Seponite.	- 202- - 202, - 208, 202- 210, - 204- 210, 	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 205 211 202 214 212
Nacrite	- 202- - 202, - 208, 202- 210, - 204- 210, 	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 205 211 202 214 212
Nacrite Nesquehonite Nontronite  Opal Oxides, hydrous  Paligorskite Penminite Phillipsite Phogopite Porters Creek clay, fuller's earth from Pitilolite Pyrophyllite Quartz, powdered  Racewinite'' Rectorite Saponite Sepicite	- 202- - 202- - 202, - 208, 202- 210, - 204- 210, 200,	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 -205 211 202 214 212 201
Nacrite Nesquehonite Nontronite  Opal Dxides, hydrous Paligorskite Penminite Phillipsite Phogopite Porters Creek clay, fuller's earth from Pitilolite Pyrophyllite Quartz, powdered Racewinite'' Rectorite Saponite Septicite Septi	- 202- - 202- - 202, - 208, 202- 210, - 204- - 200, 	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 -205 211 202 214 212 201 214
Nacrite Nesquehonite Nontronite N	- 202- - 202, - 208, 202- 210, - 204- 210, 	216 210 202 -205 212 212 203 214 212 209 -203 214 200 -205 211 202 214 212 201 214 212 212 214 212 214 212 214 214 21
Nacrite Nesquehonite Nontronite N	- 202- - 202, - 208, 202- 210, - 204- 210, 200, 197-	216 210 202 -205 212 212 203 214 212 209 -203 214 200 -205 211 202 214 212 201 214 212 212 214 212 214 212 214 214 21
Nacrite Nesquehonite Nontronite  Opal Dxides, hydrous Paligorskite Penminite Phillipsite Phogopite Porters Creek clay, fuller's earth from Pitilolite Pyrophyllite Quartz, powdered Racewinite'' Rectorite Saponite Septicite Sept	- 202- - 202- - 202, - 208, 202- 210, - 204- 210, - 197-	216 210 202 202 212 212 203 214 212 209 -203 214 200 210 -205 211 202 214 212 201 214 212 201
Nacrite Nesquehonite Nontronite  Opal Dxides, hydrous Paligorskite Penminite Phillipsite Phogopite Porters Creek clay, fuller's earth from Pitilolite Pyrophyllite Quartz, powdered Racewinite'' Rectorite Saponite Septicite Sept	- 202- - 202- - 202, - 208, 202- 210, - 204- 210, - 197-	216 210 202 202 212 212 203 214 212 209 -203 214 200 210 -205 211 202 214 212 201 214 212 201
Nacrite Nesquehonite Nontronite N	- 202- - 202, - 208, 202- 210, - 204- 210, - 200, - 197-	216 210 202 202 212 212 203 214 200 210 205 211 202 214 212 201 214 214 214 214
Nacrite Nesquehonite Nontronite N	- 202- - 202, - 208, 202- 210, - 204- 210, - 200, - 197-	216 210 202 -205 212 212 203 214 212 209 -203 214 200 210 205 211 202 214 212 201 214 214 219 214 214 219 214 219 201